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IS 13796 (2004): Molybdenum Disulphide Powder for Lubricants [PCD 3: Petroleum, Lubricants and their Related Products]

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(पहला पुनरीक्षण)

Indian Standard

MOLYBDENUM DISULPHIDE POWDER FOR LUBRICANTS — SPECIFICATION

(First Revision)

ICS 75.100

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2004

Price Group 3

Petroleum, Lubricants and Their Related Products Sectional Committee, PCD 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum, Lubricants and Their Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Molybdenum disulphide powder has become an important solid lubricants. As a boundary lubricant, it has excellent load carrying properties, low coefficient of friction and good thermal stability and chemical inertness properties. Molybdenum disulphide powder is used as oil and aqueous dispersion or compounded with greases. It is also used in oxygen valve and in pipefittings, compressor etc.

This standard was first published in 1993. In this version scope has been enlarged to cover greases for surface when boundary condition exists. Characteristics such as acid insoluble and iron content have been included.

The composition of the Committee responsible for formulation of this standard is given in Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MOLYBDENUM DISULPHIDE POWDER FOR LUBRICANTS — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for molybdenum disulphide powder intended for use as lubricants and greases for surfaces where boundary lubrication exists.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
460 (Part 1) : 1985	Specification for test sieves: Part 1 Wire cloth test sieves (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
1447 (Part 1) : 2000	Petroleum and its products— Methods of sampling : Part 1 Manual sampling (*first revision*)
1448 [P : 2] : 1967	Methods of test for petroleum and its products: [P : 2] Acidity (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be dry, free-flowing, greyish black in colour having a metallic lustre with a smooth and soapy feel. It shall be free from grit, foreign matter, grease, oil and visible impurities.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 and 5 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

Table 1 Requirements of Molybdenum Disulphide for Lubricants

(*Clause* 3.2)

Sl No.	Characteristics	Requirements	Method of Test, Ref to	
			Annex of This Standard	[P] of IS 1448
(1)	(2)	(3)	(4)	(5)
i)	Molybdenum disulphide content, percent by mass, *Min*	98.0	A	–
ii)	Sieve test:		B	–
	a) Percent passing through 150 micron Sieve, *Min*	100	–	–
	b) Percent passing through 75 micron Sieve, *Min*	99	–	–
	c) Percent passing through 63 micron Sieve, *Min*	95	–	–
iii)	Acid insolubles, *Max*	0.5	C	–
iv)	Acidity, mg of KOH/g, *Max*	0.5	–	[P : 2]
v)	Volatile matter (Evaporation loss), percent by mass, *Max*	0.1	D	–
vi)	Iron content, percent by mass, *Max*	0.3	C	–

4.2 Marking

4.2.1 Each container shall be marked with the following information:

a) Name of the material;

b) Net mass in the container;

c) Name of manufacturer, recognized trade-mark, if any, and with identification in code or otherwise to enable the lot of consignment or manufacturer to be traced back;

d) Recognized trade-mark, if any;

e) Date of manufacture; and

f) Batch No. or Code No.

4.3 BIS Certification Marking

The containers may also be marked with the Standard Mark.

4.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 1).

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MOLYBDENUM DISULPHIDE CONTENT

A-1 REAGENTS

A-1.1 Concentrated Nitric Acid, sp. gravity, 1.42 A. R. Grade.

A-1.2 Perchloric Acid, 60 percent A.R. Grade.

A-1.3 Concentrated Ammonia Solution, sp. gravity 0.880.

A-1.4 Methyl Orange Indicator, 0.5 percent (*v/v*) in industrial methylated spirit.

A-1.5 Concentrated Hydrochoric Acid, sp. gravity 1.18, A. R. Grade.

A-1.6 Ammonium Acetate Solution (Aqueous), 30 percent (*w/v*).

A-1.7 Lead Acetate Solution, 2 percent (*w/v*) in 2 percent (*v/v*) acetic acid.

A-1.8 Tannic Acid Indicator, 0.1 percent (*w/v*) in 4 percent (*v/v*) acetic acid.

A-1.9 Acetic Acid, glacial

A-2 PROCEDURE

A-2.1 Weigh approximately 0.25 g (to the nearest of 0.001 g) (W_1) of the sample into a 400 ml beaker. Add 15 ml concentrate nitric acid and 25 ml of perchloric acid. Place on hotplate and allow to fume vigorously (do not cover the beaker). Continue heating until the sample is white in colour and the volume of the acid is about 5 ml.

A-2.2 Cool and dilute to about 50 ml with distilled water. Add 40 ml of concentrate ammonia slowly with constant stirring and bring to boil.

A-2.3 Filter while hot through a 12.5 cm No. 41 Whatman filter paper. Collect the filtrate in a 600 ml beaker and wash the residue on filter paper at least 8 times with hot distilled water. The final volume of the filtrate should be approximately 400 ml.

A-2.4 Add 2-3 drops of methyl orange indicator and neutralise by the dropwise addition of concentrate hydrochloric acid with constant stirring.

A-2.5 Add 30 ml of ammonium acetate solution and 10 ml glacial acetic acid and bring almost to boil.

A-2.6 Add lead acetate solution slowly with constant stirring until an excess of 30 ml has been added. The bulk of the reagent should be added at a fixed rate of 30 ml in 2 min with mechanical stirring. Tannic acid solution is used as an external indicator, the endpoint being reached when a drop of filtrate added to a small volume of the tannic acid solution on a spot plate fails to develop a brown colouration.

A-2.7 Bring the solution to almost boiling condition and allow to remain at this temperature for 1h.

A-2.8 Filter the precipitate on to a 15 cm No. 42 Whatman filter paper and wash thoroughly with hot water.

A-2.9 Ignite the precipitate in a tared porcelain crucible at 700 °C. Cool and weigh (W_2).

A-3 CALCULATION

A-3.1 Calculate the percentage molybednum disulphide content from the following:

$$\text{Molybdenum disulphide, percent by mass} = \frac{W_2 \times 43.60}{W_1}$$

where

W_1 = mass of the sample taken, and

W_2 = mass of the ignited residue.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

METHOD FOR SIEVING TEST

B-1 APPARATUS

B-1.1 Nominal Test Sieves — Size as 150 µ, 75 µ and 63 µ, with cover and receiver [*see also* IS 460 (Part 1)].

B-2 PROCEDURE

B-2.1 Assemble the three sieves in numerical order with 150 µ size on the top and with cover and receiver.

B-2.2 Transfer approximately 5 g of the sample (weighed to 0.000 5 g) on to the top sieve.

B-2.3 Tap the sieve on a felt pad, rotating and inclining them at about 30° to the horizontal for 15 min.

B-2.4 Remove the material from the receiver pan, replace the receiver and continue with the tapping operation, the amount of material passing through sieves being noted at the end of each successive minute of tapping. The operation shall be regarded as completed when the amount passing through in 1 min does not exceed 0.01 g.

B-2.5 when sieving is completed remove the residue from each sieve by brushing it out into a tared scoop and weigh to 0.000 1 g. Calculate the percentage material, which passes through each sieve.

NOTE — It is important to note that natural molybdenum disulphide is friable and therefore, a brush cannot be used to facilitate the sieving operations.

ANNEX C

[*Table* 1, *Sl No.* (iii) *and* (vi)]

METHOD FOR THE DETERMINATION OF ACID INSOLUBLES AND IRON CONTENT

C-1 SCOPE

The method describes a procedure for the determination of acid insoluble matter and iron.

C-2 OUTLINE OF METHOD

After oxidation of the sample with nitric acid and potassium chlorate the insoluble matter is filtered and determined gravimetrically. The iron content is determined by spectrometric analysis of the filtrate.

C-3 APPARATUS

C-3.1 Beakers, Squat Form, with lip 600 ml.

C-3.2 Filter Funnel, Buchner, 55-70 mm diameter.

C-3.3 Filter Papers, Quantitative, Whatman No. 542, 55-70 mm.

C-3.4 Measuring Cylinders, 10 ml and 250 ml.

C-3.5 Muffle Furnace, controlled at 600 ± 10 °C.

C-3.6 Crucible, Silica, Fused, to accommodate the filter papers.

C-3.7 Volumetric Flasks, 25 ml and 250 ml.

C-3.8 Pipettes, 5 ml.

C-3.9 Hotplate

C-3.10 Water Bath

C-3.11 Atomic Absorption Spectrophotometer

C-3.12 Desiccator with Desiccant

C-4 REAGENTS

C-4.1 Water, *see* IS 1070.

C-4.2 Nitric Acid, concentrated analytical reagent grade density 1.42.

C-4.3 Nitric Acid, dilute 20 percent (*v*/*v*) aqueous grade.

C-4.4 Potassium Chlorate, analytical reagent grade.

C-4.5 Hydrochloric Acid, analytical reagent grade, density 1.18.

C-4.6 Hydrochloric Acid, 50 percent (*v/v*) aqueous solution of the above.

C-4.7 Ammonium Persulphate, analytical reagent grade.

C-4.8 Ammonia Solution, density 0.88.

C-4.9 Ammonium Carbonate, analytical reagent grade, 2 percent *m/v* aqueous solution.

C-4.10 Standard Iron Solutions, aqueous: Blank, 2.5, 5.0, 10.0, 15.0, 20.0 µg / 1 Fe.

NOTE — BDH spectrosol standard have been found to be satisfactory.

C-5 PROCEDURE

C-5.1 A blank determination (that is omitting the sample) shall be conducted alongside the sample.

C-5.2 Weigh 5 ± 0.05 g to the nearest 0.01 g of sample into a 600 ml beaker. Add 15 ml of water, 10 ml of the concentrated nitric acid density 1.42, and bring to a gentle boil on the hotplate.

C-5.3 Remove from heat and after 10 min add 10 ml of the concentrated nitric acid density 1.42 reheat and remove from the hotplate to allow the reaction to subside. Add a further 10 ml of the nitric acid and heat (do not heat to dryness) until the mixture assumes a grey colouration, then remove from heat.

C-5.4 Add 25 ml of the concentrated nitric acid density 1.42 and bring to the boil. Add small quantities (0.25-0.5 g) of the potassium chlorate whilst swirling until full oxidation has taken place as noted by the appearance of a creamy white colouration. Add a further 2 g of the potassium chlorate with swirling and evaporate gently on the water bath until just dry. Do not overheat or bake the residue. Remove and cool to room temperature.

C-5.5 To the contents of the beaker add 50 ml of the hydrochloric acid density 1.18 and boil until solids are in solution. Add 2 g of the ammonium persulphate, boil to reduce the volume to about 100 ml, allow to cool to room temperature and any solid to settle out.

C-5.6 Filter the supernatent liquid through a Whatman No. 542 paper in the filter funnel using gentle suction. Rinse the beaker and filter five times with the warm, ammonium carbonate solution, followed by two rinsings of hot water. Discard the filtrate.

C-5.7 Dissolve the iron from the residue with three washes of hot 50 percent hydrochloric acid, followed by hot water, until the volume of filtrate is about 200 ml. Retain the filtrate for the iron determination.

C-5.8 Transfer the filter papers to a silica crucible previously ignited at 600 °C, and tared to 0.001g. Carefully burn off the paper and place the crucible in the muffle furnace at 600 ± 10°C for 1 h. Allow the crucible to cool in a desiccator.

C-5.9 Weigh the crucibles and the contents to the nearest 0.001 g.

C-6 DETERMINATION OF IRON

C-6.1 Procedure

C-6.2 Quantitatively transfers the filtrate obtained in C-5 to a 250 ml volumetric flask and adjust to volume with the water.

C-6.3 Pipette 5 ml of the solution into a 25 ml volumetric flask, add 4 ml of the 50 percent hydrochloric acid, 2 ml of the 20 percent nitric acid and adjust to volume with the water.

C-6.4 Optimize the atomic absorption spectrophotometer at the following parameters:

a) Wavelength : 302.1 nm
b) Band width : 0.3 nm
c) Lamp current : 3A
d) Background : For example Deuterium lamp
e) Flame : Air-acetylene oxidizing lean blue.

C-6.5 Aspirate the standard iron solutions in sequence through the flame and plot a graph of absorbance against concentration.

C-6.6 Aspirate the blank and sample filtrates in sequence and note the absorbance values.

C-7 CALCULATION

C-7.1 Calculate the acid insoluble matter to the nearest 0.01 percent as follows:

$$\text{Acid insoluble matter, percent } m/m = \frac{a - b \times 100}{\text{Mass of sample}}$$

where

a = mass of residue from the sample, and
b = mass of residue from the blank.

C-7.2 Calculate the iron content to the nearest 0.01 from the graph produced by the standard solutions, taking into account the iron concentration in the blank.

C-8 REPORTING

C-8.1 Report the acid insoluble matter to the nearest 0.001 percent.

C-8.2 Report the iron concentration to the nearest 0.01 percent.

C-9 PRECISION

To be determined.

ANNEX D

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF VOLATILE MATTER

D-1 APPARATUS

D-1.1 Electric Oven with a facility to maintain the temperature at 105 ± 2°C

D-1.2 Electrical/Electronic Balance to weigh the samples up to 10 g with an accuracy of 0.001g.

D-1.3 Glass Dishes

D-1.4 Desiccator

D-2 PROCEDURE

D-2.1 Weigh approximately 5 g (to nearest of 0.001 g) of the sample into the glass dish (approximately 50 mm diameter and 30 mm in height) fitted with a ground glass lid.

D-2.2 Transfer the dish to an oven maintained at 105 ± 2°C and remove the lid. Allow the sample to remain in the oven for 4 h.

D-2.3 At the end of 4 h remove the dish to a desiccator. Cool and replace the lid and re-weigh.

D-3 CALCULATION

D-3.1 Record the loss in weight of the sample and calculate as percentage volatile matter.

$$\text{Volatile content, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass of sample before heating, and
M_2 = mass of sample after heating.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Petroleum Lubricants and Their Related Products Sectional Committee, PCD 3

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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